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(54) **Water-in-oil emulsions and process for preparing same.**

(57) A high internal phase water-in-oil emulsion comprises, in addition to water, a branched chain non-polar oil, a liquid emulsifier having an HLB value of from 1 to 7 and a special montmorillonite clay derivative which stabilises the emulsion. The emulsion can optionally also include a source of magnesium ions which further enhance its stability during storage. The emulsion is particularly suitable as a cosmetic cream for topical application to the skin.

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**TITLE MODIFIED**  
see front page.

WATER-IN-OIL EMULSIONS

The invention relates to emulsions. More particularly, the invention relates to high internal phase water-in-oil emulsions suitable for the preparation of cosmetic, pharmaceutical and other products and to methods for the  
5 preparation of such emulsions.

By "high internal phase emulsion" is meant an emulsion in which the volume of the internal phase occupies at least 75% of the total volume of the emulsion.

Water-in-oil emulsions, other than high internal phase  
10 emulsions, have been employed in the formulation of cosmetic and pharmaceutical ointments, emollient creams, lotions and the like. However, while such emulsions can impart desirable characteristics of water-repellancy to the skin and provide a means whereby fats, oils and waxes can be  
15 absorbed onto human skin, the use of such emulsions in skin

treatment products has sometimes been precluded because they have been implicated in toxicity, skin irritancy or excessive greasiness in use. The problem of instability has also limited the used of high internal phase water-in-oil emulsions in cosmetics and pharmaceutical products where storage stability is a pre-requisite.

A study was accordingly undertaken to examine the instability of these emulsions and to establish means whereby stability could be improved. It was found, for example, that high internal phase water-in-oil emulsions containing as skin benefit agents a water soluble salt such as an organic lactate or a metallic sulphate dissolved in the aqueous phase, are stable for no more than a few days. Syneresis of the internal phase or even separation of the oily and aqueous phases can then generally be seen as an accumulation of oil at the surface of the emulsion.

This problem has led to the screening of many substances in a search for a stabilising agent whose incorporation in a product based on a high internal phase emulsion would effectively extend the shelf-life of that product at normal storage temperatures.

It has now been discovered that this problem of instability of the high internal phase emulsion on storage can be overcome by incorporation into the emulsion of a special montmorillonite clay derivative, and optionally a water-soluble salt. The high internal phase emulsions so obtained have good storage characteristics in that the water and oil phases do not separate when the emulsion is stored for at least three months, even at a "tropical" temperature of 50°C, or when subjected repeatedly to freezing at a temperature of -20°C and thawing at ambient temperature of +20°C.

These high internal phase emulsions are accordingly suitable for use in the preparation of cosmetic and pharmaceutical products and the like, particularly for

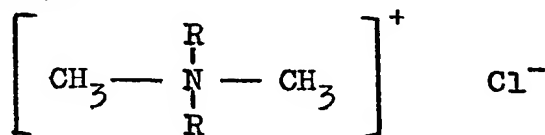
topical application to human skin, which avoid the aforementioned disadvantages in that they are non-toxic, non-irritating to the skin, not excessively greasy in use and which do not suffer from instability on storage.

5 It has also been observed that inclusion of the special montmorillonite clay derivative in the emulsion unexpectedly provides a further benefit in addition to that of imparting a very high degree of stability to the emulsion after manufacture and during storage, and that  
10 it is enabling the emulsion to "break" almost instantly when applied topically to human skin, thus enabling any skin benefit ingredient in the emulsion to be more readily available for uptake by the skin.

The stability imparted by the special montmorillonite  
15 clay derivative is all the more surprising when it is realised how intrinsically unstable are high internal phase water-in-oil emulsions. It is accordingly totally unexpected that the special montmorillonite clay derivative should exhibit, as we have shown, on the one hand stability of the  
20 emulsion in its container, while on the other hand allowing the emulsion to "break" when applied topically to the skin.

Accordingly, the invention provides a high internal phase water-in-oil emulsion, characterised in that it comprises in addition to water:

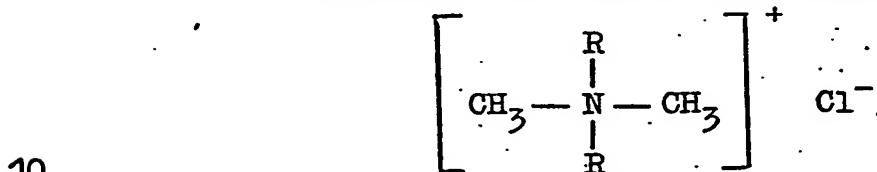
- 25 (a) a branched chain non-polar oil;  
(b) a liquid emulsifier having an HLB value of from 1 to 7; and  
(c) a reaction product of a sodium magnesium-fluorolithosilicate trioctahedral montmorillonite  
30 clay and the quarternary ammonium salt having the formula:



where R represents hydrogenated tallow fatty acid radicals.

The invention also provides a process for preparing a high internal phase water-in-oil emulsion which comprises the steps of:

- 5 (a) mixing the reaction product of a sodium magnesium-fluorolithosilicate trioctahedral montmorillonite clay and the quaternary ammonium salt having the formula:



- where R represents hydrogenated tallow fatty acid radicals with a liquid emulsifier having an HLB value of from 1 to 7 and a branched chain non-polar oil to provide an oily phase; and
- 15 (b) homogenising the oily phase with an aqueous phase comprising water and water soluble ingredients to provide a water-in-oil emulsion in which the aqueous phase forms from 75 to 98% by volume of the emulsion and the oily phase forms from 2 to
- 20 25% by volume of the emulsion.

The emulsion according to the invention consists of an internal phase which is aqueous and an external phase which is oily. It should be explained that water-in-oil emulsions which are not high internal phase emulsions

25 usually consist of from about 1 to 74% by volume of an aqueous phase dispersed in about 99 to 26% by volume of an oily phase. A water-in-oil emulsion consisting of 74% by volume aqueous phase and 26% by volume oily phase represents the theoretical maximum packing volume

30 concentration of rigid monodisperse spheres of water as the internal phase in oil as the external phase. Hence, water-in-oil emulsions containing more than 74% aqueous phase are high internal phase emulsions.

The water-in-oil emulsion of the invention comprises

35 from 75 to 98%, preferably 80 to 97% by volume of an

aqueous phase and from 2 to 25%, preferably 3 to 20% by volume of an oily phase respectively.

In order to obtain stable high internal phase water-in-oil emulsions, it has been shown necessary to select carefully the oil ingredient, the emulsifier and to employ a special montmorillonite clay derivative as a special emulsion stabiliser to ensure that the emulsions once made generally remain stable over an extended period of time.

#### The Oil

The oil should preferably be liquid at room temperature (20°C) and should be cosmetically and pharmaceutically acceptable. It is however also possible to employ waxes which can be solid at room temperature. The oil, other oily material and wax are herein referred to as "oil".

The oil should also be non-polar and should contain branched chain alkyl groups. The preferred oils are highly branched-chain mineral oils.

Examples of preferred oils are (in decreasing order of preference):

C<sub>10</sub> to C<sub>12</sub> isoparaffins such as ISOPAR L (Esso)

Polyisobutene such as PARLEAM (Nichiyu)

Squalane such as COSBIOL (Laserson & Sabetay)

Branched chain light paraffin oil such as LYTOL (Witko) or WML (BP)

Mineral oil such as MARCHOL 82 (Esso) or CARNATION OIL (Witko)

Petrolatum such as VASELINE (Gerland)

Microcrystalline wax such as CEREWAX L (La Ceresine)

Lanolin derivatives such as MODULAN (Amerchol)

Oleic decylester such as CETIOL V (Henkel)

Ethyl hexylpalmitate such as WICKENOL 155

C<sub>16</sub> to C<sub>18</sub> fatty alcohol di-isooctanoate such as CETIOL SN (Henkel)

It is also possible to employ vegetable and animal oils, provided that branched-chain alkyl groups are present.

The quantity of oil in the emulsion is from 1 to 24%, preferably from 2 to 15% by weight of the emulsion.



If the emulsion contains less than 1% oil, it is generally not possible to obtain a stable water-in-oil emulsion, whereas if the emulsion contains more than 24% oil, then the emulsion will cease to exhibit the special properties and characteristics attributable to a high internal phase emulsion.

#### The Emulsifier

The emulsifier should be liquid at room temperature (20°C) and be cosmetically and pharmaceutically acceptable.

The emulsifier should have an HLB value of from 1 to 7, preferably from 2 to 6.

Examples of suitable emulsifiers are (in decreasing order of preference):

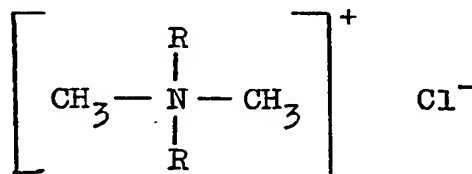
		<u>HLB Value</u>
	ARLACEL 987 (sorbitan isostearate) by Atlas	7
15	HOSTAPHAT KO 300N (mono-, di-, and tri-phosphoric esters of oleic acid) by Hoechst	2.3
	IMWITOR 780K (glycerol monoisostearate) by Witco	3.7
20	BRIJ 92 (polyoxyethylene(2)oleyl ether) by Atlas	4.9
	Triglycerol monooleate by PVO International	4.0
	ARLACEL 80 (sorbitan monooleate) by Atlas	4.3
	ARLACEL 83 (sorbitan sesquioleate) by Atlas	7
	ARLACEL 85 (sorbitan trioleate) by Atlas	1.8
25	Decaglycerol tetraoleate by PVO International	6.0
	Decaglycerol octaoleate by PVO International	4.0
	SIMULSOL 92 (polyethoxylated(2)oleyl alcohol) by Produits Chimiques de la Montagne Noire	6.7

The quantity of emulsifier in the emulsion is from 0.5 to 10%, preferably 2 to 5% by weight of the emulsion.

If the emulsion contains less than 0.5% of emulsifier, it is unlikely that the emulsion, if obtained, will remain stable on storage, whereas if the emulsion contains more than 10% of emulsifier, the stability of the emulsion can be adversely affected.

The special montmorillonite clay derivative

The special montmorillonite clay derivative is a reaction product of a sodium magnesium - fluorolithosilicate trioctahedral montmorillonite clay and the quaternary ammonium salt having the formula:



where R represents hydrogenated tallow fatty acid radicals.

This montmorillonite clay derivative is hereinafter referred to as Quarternium -18 Hectorite. An example of Quarternium -18 Hectorite is BENTONE 38 available from National Lead Industries.

The quantity of Quarternium -18 Hectorite in the emulsion is from 0.1 to 10%, preferably 0.1 to 1% by weight of the emulsion.

If the emulsion contains less than 0.1% of the Quarternium -18 Hectorite, it is unlikely that the emulsion will remain stable on storage, whereas if the emulsion contains more than 10% Quarternium -18 Hectorite, the emulsion is likely to be too viscous to employ as a cosmetic or pharmaceutical product, particularly for topical application to the skin.

Water

The emulsion also comprises water. The quantity of water in the emulsion is from 0.1 to 97%, preferably 60 to 95% by weight of the emulsion.

If the emulsion contains more than 97% of water, the stability of the emulsion on storage is likely to be poor and syneresis can occur.

Optional emulsion stabilising ingredients

The emulsion of the invention can optionally also comprise a source of magnesium ions which can serve not only to provide an additional cosmetic benefit to the emulsion when applied topically to human skin, but also to further

enhance the stability of the emulsion during storage after manufacture and before use.

5 Magnesium ions can preferably be provided by incorporating into the emulsion a water soluble salt of magnesium, such as magnesium sulphate.

The quantity of magnesium sulphate in the form of the heptahydrate that can be incorporated in the emulsion is from 0.1 to 5%, preferably 0.1 to 2% by weight of the emulsion.

10 If the emulsion contains less than 0.1% of magnesium sulphate heptahydrate, it is unlikely that the stability of the emulsion will be improved beyond that due to the presence of the Quarternium -18 Hectorite, whereas of the emulsion contains more than 5% magnesium sulphate heptahydrate,  
15 it is unlikely that the stabilisation of the emulsion can be further enhanced.

#### Cosmetically and Pharmaceutically Active Ingredients

The emulsion according to the invention can be employed as a vehicle for a wide variety of cosmetically or  
20 pharmaceutically active ingredients, particularly ingredients which have some beneficial effect when applied to the skin.

The emulsion thus provides a means whereby such active ingredients can be diluted, preserved, conveyed to and distributed on the skin surface at an appropriate  
25 concentration.

Especially preferred examples of active ingredients include moisturisers such as: sodium pyrrolidone carboxylate, sodium lactate, lactic acid, triethanolamine lactate and sodium chloride.

30 Examples of other active ingredients that can also be employed include sunscreen agents, germicides, deodorants, antiperspirants, healing agents.

#### Functional Adjuncts

35 The emulsion according to the invention can also contain functional adjuncts for further controlling the properties of a pharmaceutical or cosmetic composition

containing the emulsion. Functional adjuncts include: antioxidants, propellants, solvents, humectants, thickeners and emollients.

Preparation of emulsion

5       The high internal phase water-in-oil emulsions of the invention can be prepared by mixing the Quarternium -18 Hectorite with the liquid emulsifier having an HLB value of from 1 to 7 and the oil to provide an oily phase and subsequently homogenising from 2 to 25 parts by volume of  
10       the oily phase with from 75 to 98 parts by volume of the aqueous phase water and water soluble ingredients to provide the emulsion.

      The oily phase so prepared will comprise from 0.1 to 10 parts by weight of the Quarternium -18 Hectorite, from  
15       0.5 to 10 parts by weight of the liquid emulsifier and from 1 to 24 parts by weight of the oil.

      The aqueous phase will comprise not more than 97 parts by weight of water.

      It is to be noted that the Quarternium -18 Hectorite  
20       will not normally impart any discernable thickening to the oily phase alone but after emulsification with the aqueous phase, the viscosity of the emulsion will increase to provide the stabilised emulsion.

Product Forms

25       The compositions of the invention can be formulated as liquid, for example products such as lotions for use in conjunction with applicators such as a roll-ball applicator or a spray device such as an aerosol can containing propellant or a container fitted with a pump to dispense  
30       the product. Alternatively, the compositions of the invention can be solid or semi-solid, for example powders, moulded sticks, creams or gels, for use in conjunction with an applicator such as a powder sifter or a stick applicator, or simply a tube or lidded jar.

35       The invention accordingly also provides a closed

container containing a cosmetically acceptable composition as herein defined.

The invention is illustrated by the following examples:

5

Example 1

This example illustrates a moisturising cream for topical application to the skin.

This cream has the following formulation:

		<u>% W/W</u>
	(a) <u>branched chain non polar oils:</u>	
10	(i) CARNATION OIL, (a light liquid paraffin available from Witco)	5.0
	(ii) Oleic acid decylester (CETIOL V available from Henkel)	5.0
	(b) <u>a liquid emulsifier having an HLB</u>	
15	<u>value of from 1 to 7:</u>	
	Sorbitan isostearate (ARLACEL 987 available from Atlas)	2.5
	(c) <u>Quarternium -18 Hectorite:</u>	
20	BENTONE 38 (available from National Lead Industries)	0.5
	(d) <u>Magnesium Sulphate:</u>	
	MgSO <sub>4</sub> ·7H <sub>2</sub> O	0.3
	(e) Emollient, moisturising electrolyte, preservations and perfume	13.6
25	(f) <u>Water:</u>	
	deionised water	78.1

The oils were mixed with the emulsifier, the Quarternium -18 Hectorite and the preservative to provide the oily phase. The aqueous phase containing water, the  
30 magnesium sulphate and the emollient and moisturising electrolyte was then added slowly at first and then more rapidly to the oily phase with stirring until an emulsion in the form of a white cream was obtained. The  
35 mixing step was carried out at room temperature, i.e. at about 23°C. Finally the perfume was distributed throughout the emulsion.

In order to test the stability of the cream, samples were stored at 23°C, 35°C, 42°C and at 50°C. After more than six months' storage, there was no sign in any of the samples of syneresis or breakdown of the emulsion.

- 5 Likewise, repeated freeze-thaw cycling from -5°C to +42°C did not indicate any instability in the product.

The product was accordingly judged completely stable and suitable for sale to consumers.

- 10 When applied to the human skin, the product has a pleasant creamy texture, is easily distributed topically and "breaks" on contact with the skin. The release of the aqueous phase gives the consumer a pleasant fresh sensation. which is judged as signalling a moisturizing effect.

Examples 2&3

Examples 2 and 3 illustrate moisturising lotions.

		<u>% w/w</u>	
		<u>Ex.2</u>	<u>Ex.3</u>
5	HOSTAPHAT KO 300 N	3	-
	IMWITOR 780K	-	3
	ISOPAR L	15	15
	BENTONE 38	0.3	0.4
	Triethanolamine lactate - 50%:pH 5.5		
10	(moisturising agent)	6	6
	Para P <sup>(1)</sup>	0.1	0.1
	1,3-butylene glycol	3	3
	Water	72.6	72.5

(1) propyl p-hydroxybenzoate.

- 15 The oil is mixed with the emulsifier, the Para P added and dissolved at a temperature of 60-70°C. The BENTONE 38 is then added to provide the oily phase. The aqueous phase containing the lactate and the glycol are heated gently at 45-50°C. Finally, the emulsion is prepared
- 20 after cooling by emulsifying the aqueous phase with the oily phase to provide a water-in-oil emulsion at a temperature no higher than 50°C.

Example 4

- 25 An emulsion having the following formulation as a further example of a skin moisturising lotion for topical application is prepared by the method described for Examples 2 and 3. It contains the following ingredients:

<u>Ingredients</u>		<u>% w/w</u>
5	O { HOSTAPHAT KO 300 N - ex Hoechst	3
	LYTOL - ex Witco	15
	Para P - ex Rhone Poulenc	0.1
	BENTONE 38	0.5
10	A { Sodium pyrrolidone carboxylate (Na PC)	
	(moisturising agent) (50% in water)	4
	1,3-butylene glycol	3
	Collagen hydrolysate	3
	Para M	0.2
	Water	71
	Perfume	0.2

O - Oily phase

A - Aqueous phase

15

Examples 5 to 7

These emulsions illustrate the formulation of moisturising creams for topical application to the skin:



	Example 5	Example 6	Example 7
(	3	3	3
HOSTAPHAT KO 300 N - ex Hoechst			
(	5	-	-
ISOPAR L - ex Esso			
(	5	-	5
PARLEAM - ex Nichiyu			
(	-	10	-
LYTOL - ex Witco			
(	-	-	5
WVI oil - ex BP			
(	0.1	0.1	0.1
PARA P - ex Rhone Poulenc			
(	0.6	0.8	1.0
BENTONE 38			
(	4	4	4
Na PC (50%)			
(	3	3	3
1,3-butylene glycol			
(	3	3	3
Collagen hydrollysate			
(	-	2	2
Na glutamate			
(	0.2	0.2	0.2
PARA M - ex Rhone Poulenc			
(	75.9	73.7	73.5
Water			
(	0.2	0.2	0.2
Perfume			

0

5

10

A

15

Example 8

This example also illustrates the formulation of a moisturising cream for topical application to the skin:

	<u>Ingredients</u>	<u>% w/w</u>
5	( HOSTAPHAT KO 300 N	3
	( LYTOL	5
	( MARCHOL 82	5
0	( CEREWAX L	0.2
	( MODULAN	1
10	( PARA P	1
	( BENTONE 38	2
	( Na PC (50%)	4
	( 1,3-butylene glycol	3
	( Collagen hydrolysate	3
15	A ( Na glutamate	1
	( PARA P	0.2
	( Water	71.3
	( Perfume	0.2

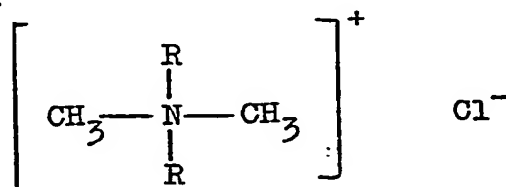
Example 9

This example also illustrates the formulation of a moisturising cream for topical application to the skin:

	<u>Ingredients</u>	<u>% w/w</u>
	( HOSTAPHAT KO 300 N	3.5
	( ISOPAR L	5
25	( PARLEAM	5
	( CEREWAX L	0.2
	( MODULAN	1
	( PARA P	0.1
	( BENTONE 38	1.5
30	( Na PC (50%)	4
	( Glycine	5
	A ( PARA M	0.2
	( Water	71.3
	( Perfume	0.2

CLAIMS

1. A high internal phase water-in-oil emulsion, characterised in that it comprises in addition to water:
- (a) a branched chain non-polar oil;
  - (b) a liquid emulsifier having an HLB value of from 1 to 7; and
  - (c) a reaction product of a sodium magnesium - fluorolithosilicate trioctahedral montmorillonite clay and the quarternary ammonium salt having the formula:



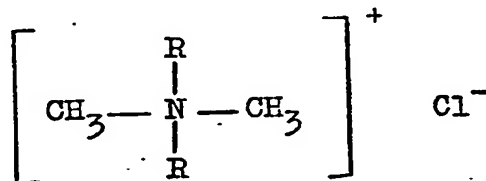
where R represents hydrogenated tallow fatty acid radicals.

2. An emulsion according to claim 1, characterised in that the reaction product of the sodium magnesium - fluorolithosilicate trioctahedral montmorillonite clay and the quarternary ammonium salt forms from 0.1 to 10% by weight of the emulsion.
3. An emulsion according to claim 1, characterised in that the reaction product of the sodium magnesium - fluorolithosilicate trioctahedral montmorillonite clay and the quarternary ammonium salt forms from 0.1 to 1.0% by weight of the emulsion.
4. An emulsion according to claim 1, 2 or 3, characterised in that the water forms from 75 to 97% by weight of the emulsion.
5. An emulsion according to any preceding claim, characterised in that the oil forms from 1 to 24% by weight of the emulsion.

6. An emulsion according to any preceding claim, characterised in that the emulsifier forms from 0.5 to 10% by weight of the emulsion.
7. An emulsion according to any preceding claim, characterised in that it comprises from 75 to 98% by volume of an aqueous phase.
8. An emulsion according to any preceding claim, characterised in that it comprises from 80 to 97% by volume of an aqueous phase.
9. An emulsion according to any preceding claim, characterised in that it comprises from 2 to 25% by volume of an oily phase.
10. An emulsion according to any preceding claim, characterised in that it comprises from 3 to 20% by volume of an oily phase.
11. An emulsion according to any preceding claim, characterised in that it additionally comprises magnesium ions.
12. An emulsion according to claim 11, characterised in that the magnesium ions are derived from magnesium sulphate.
13. An emulsion according to claim 12, which comprises from 0.1 to 5% by weight of magnesium sulphate hepta hydrate.
14. An emulsion according to claim 12 and 13, which comprises from 0.1 to 1% by weight of magnesium sulphate hepta hydrate.

15. An emulsion according to any preceding claim, characterised in that it comprises by weight of the emulsion:

- (a) from 2 to 15% of a branched chain non-polar oil;
- (b) from 2 to 5% of a liquid emulsifier having an HLB value of from 1 to 7;
- (c) from 0.1 to 10% of a reaction product of a sodium magnesium - fluorolithosilicate trioctahedral montmorillonite clay and the quarternary ammonium salt having the formula:



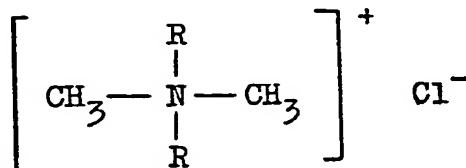
where R represents hydrogenated tallow fatty acid radicals;

- (d) from 0.1 to 5% of magnesium sulphate hepta hydrate; and
- (e) from 60 to 95% of water.

16. An emulsion according to any preceding claim and substantially as described in any of the Examples.

17. A process for preparing a water-in-oil emulsion according to any preceding claim, characterised in that it comprises the steps of:

- (a) mixing the reaction product of a sodium magnesium - fluorolithosilicate trioctahedral montmorillonite clay and the quarternary ammonium salt having the formula:



where R represents hydrogenated tallow fatty

- (b) acid radicals with a liquid emulsifier having an HLB value of from 1 to 7 and a branched chain non-polar oil to provide an oily phase; and homogenising the oily phase with an aqueous phase comprising water and water soluble ingredients to provide a water-in-oil emulsion in which the aqueous phase forms from 75 to 98% by volume of the emulsion and the oily phase forms from 2 to 25% by volume of the emulsion.



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# EUROPEAN SEARCH REPORT

**0047804**  
Application number  
EP 80 30 3230

DOCUMENTS CONSIDERED TO BE RELEVANT			CLASSIFICATION OF THE APPLICATION (Int. Cl.)
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	
X	<p><u>EP - A - 0 009 404</u> (UNILEVER)</p> <p>* The whole document, especially page 16, table 2a, examples P4, P5; page 32, examples 12,13; page 34, example 16 *</p> <p>---</p>	1-17	A 61 K 7/48 9/10
X	<p><u>FR - A - 2 409 081</u> (PETERSON/PURITAN)</p> <p>* Page 1, lines 1-6; page 2, line 29 - page 5, line 26; page 6, line 2 - page 7, line 15, pages 13-16; examples 5-7; claims 1-5, 21 *</p> <p>&amp; GB - A - 2 009 617</p> <p>---</p> <p>H. JANISTYN: "Handbuch der Kosmetika und Riechstoffe", 3rd edition, vol. 1, 1978 "Die kosmetischen Grundstoffe" Dr. Alfred, Hüthig Verlag, HEIDELBERG (DE)</p> <p>* Pages 123, 124; Bentone; pages 617-618 Miglyol-Gel *</p> <p>---</p>	1-17	TECHNICAL FIELDS SEARCHED (Int. Cl.)
		1-3	A 61 K 7/48 9/10
A	<p><u>GB - A - 883 254</u> (ABOTT LAB.)</p> <p>* Page 2, line 1 - page 3, line 2; page 3, lines 88-109 *</p> <p>---</p>		CATEGORY OF CITED DOCUMENTS
A	<p><u>FR - A - 2 419 758</u> (SHISEIDO)</p> <p>* Page 2, lines 10-15; pages 10-11, example 13 *</p> <p>-----</p>		<p>X: particularly relevant</p> <p>A: technological background</p> <p>O: non-written disclosure</p> <p>P: intermediate document</p> <p>T: theory or principle underlying the invention</p> <p>E: conflicting application</p> <p>D: document cited in the application</p> <p>L: citation for other reasons</p>
<p>The present search report has been drawn up for all claims</p>			<p>&amp;: member of the same patent family.</p> <p>corresponding document</p>
Place of search		Date of completion of the search	Examiner
The Hague		28-04-1981	BENZ

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